

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date  
25 March 2004 (25.03.2004)

PCT

(10) International Publication Number  
**WO 2004/024304 A2**

(51) International Patent Classification<sup>7</sup>: **B01D 65/02**

Ibaraki, Ibaraki 300-0315 (JP). **GOTO, Takumi** [JP/JP]; 1-5-1, Nishigotanda, Shinagawa-ku, Tokyo, Tokyo 141-0031 (JP). **HURWITZ, Mark, F.** [US/US]; 345 Snyderhill Road, Ithaca, NY 14850 (US).

(21) International Application Number:  
PCT/US2003/027655

(74) Agents: **JAY, Jeremy, M.** et al.; **LEYDIG, VOIT & MAYER**, 700 Thirteenth Street, NW, Suite 300, Washington, DC 20005 (US).

(22) International Filing Date:  
4 September 2003 (04.09.2003)

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

(25) Filing Language: English

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(26) Publication Language: English

(30) Priority Data:  
60/410,274 13 September 2002 (13.09.2002) US

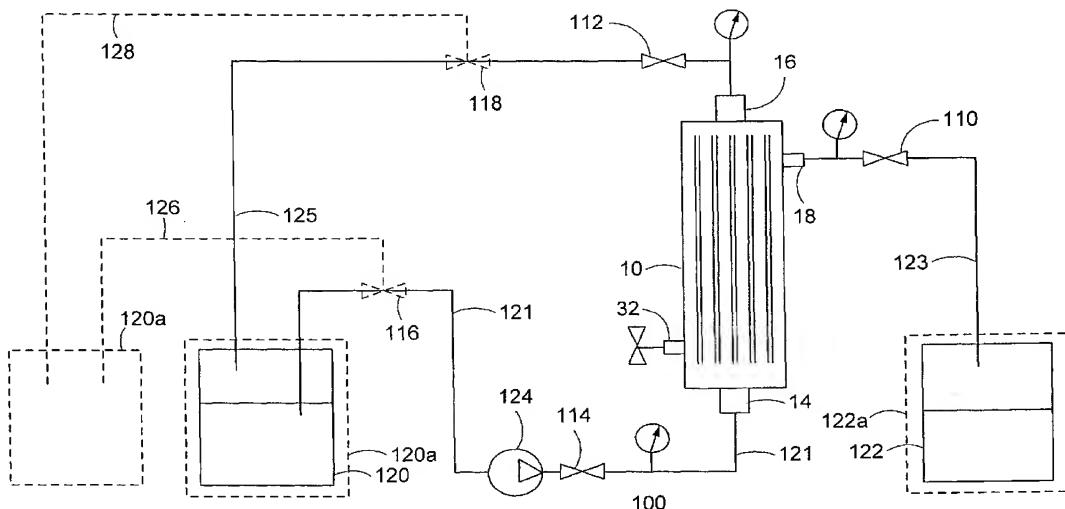
(71) Applicant (*for all designated States except US*): **PALL CORPORATION** [US/US]; 2200 Northern Boulevard, East Hills, NY 11548-1209 (US).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **AOYAMA, Kazumi** [JP/JP]; 46, Kasuminosato, Ami-machi, Inashiki-gun, Ibaraki, Ibaraki 300-0315 (JP). **KURASHIMA, Hideki** [JP/JP]; 46, Kasuminosato, Ami-machi, Inashiki-gun,

*[Continued on next page]*

(54) Title: SYSTEMS AND METHODS FOR CLEANING HOLLOW FIBER MEMBRANES



(57) Abstract: A method of cleaning hollow fiber membranes comprising directing a fluid into a hollow fiber filtration module (10) comprising a housing (12), a feed fluid inlet (14), a retentate outlet (16), a permeate outlet (18), and at least one hollow fiber membrane (20) disposed in the housing (12), wherein the hollow fiber membrane (20) has a bore (26), an inner surface (28), and an outer surface (30), providing a first pressure to the inner surface (28) of the hollow fiber membrane (20) through the retentate outlet (16) and passing the fluid along the bore (26) of at least one hollow fiber membrane (20) to provide a retentate and a permeate, increasing the first pressure, applying a second pressure to the outer surface (30) of the hollow fiber membrane (20) through the permeate outlet (18), and releasing the increased first pressure while maintaining or increasing the second pressure to pass permeate from the outer surface (30) to the inner surface (28) of the hollow fiber membrane (20), is disclosed.

WO 2004/024304 A2



**Published:**

- without international search report and to be republished upon receipt of that report

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

## SYSTEMS AND METHODS FOR CLEANING HOLLOW FIBER MEMBRANES

## CROSS-REFERENCE TO RELATED PATENT APPLICATIONS

[0001] This patent application claims the benefit of U.S. Provisional Patent Application No. 60/410,274 filed September 13, 2002, which is incorporated herein by reference.

## FIELD OF THE INVENTION

[0002] This invention pertains to cleaning hollow fiber membranes, particularly reverse cleaning of hollow fiber membranes.

## BACKGROUND OF THE INVENTION

[0003] Hollow fiber membranes are used in a variety of separation applications. For example, in a typical tangential flow filtration (TFF) application, a hollow fiber filtration module comprising a housing including a feed fluid inlet, a permeate outlet, a retentate outlet, and a plurality of hollow fiber membranes disposed in the housing, is utilized. A feed fluid is directed, via a pump, along the bores of the plurality of hollow fiber membranes, each membrane having an inner surface and an outer surface, such that a permeate or filtrate passes from the inner surface to the outer surface and a retentate passes tangentially to the inner surface and exits the bores without passing to the outer surface. However, some substances in the feed fluid accumulate in and/or on the inner surface of the membranes (i.e., a process known as fouling). The accumulation can affect the separation efficiency of the membranes and/or result in an increased pressure differential across the membranes requiring an increase in pressure to maintain a desired flow of permeate.

[0004] In some hollow fiber membrane systems, a reverse cleaning circuit comprising a separate pump, and at least one conduit communicating with a port on the module housing, is used to introduce a cleaning fluid into the module, and the cleaning fluid is directed to flow in a direction opposite to the flow of permeate through the membrane. This reverse flow (sometimes referred to as backwashing) lifts and removes substances disposed on or trapped within the membrane.

[0005] However, these systems and methods suffer from a number of drawbacks. For example, a reverse cleaning circuit utilizes an additional pump, as well as additional fluid lines, ports, and valves, increasing the complexity and/or size of the overall system.

[0006] The present invention provides for ameliorating at least some of the disadvantages of the prior art. These and other advantages of the present invention will be apparent from the description as set forth below.

### BRIEF SUMMARY OF THE INVENTION

[0007] In an embodiment, the invention provides a method of cleaning hollow fiber membranes comprising (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, wherein the hollow fiber membrane has a bore, an inner surface, and an outer surface, (2) providing a first pressure to the inner surface of the hollow fiber membrane through the retentate outlet and passing the fluid along the bore of at least one hollow fiber membrane to provide a retentate and a permeate, (3) increasing the first pressure, (4) applying a second pressure to the outer surface of the hollow fiber membrane through the permeate outlet, and (5) releasing the increased first pressure while maintaining or increasing the second pressure to pass permeate from the outer surface to the inner surface of the hollow fiber membrane.

[0008] In another embodiment of the invention, a method of cleaning hollow fiber membranes comprises directing fluid into a hollow fiber filtration module including a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least two hollow fiber membranes, wherein each hollow fiber membrane has a bore, an inner surface, and an outer surface, passing fluid into the bores of at least two hollow fiber membranes to provide retentate flow along the inner surfaces of the membranes and permeate flow from the inner surfaces of the membranes to the outer surfaces, blocking the permeate flow, restricting the retentate flow, and unrestricting the retentate flow to reverse permeate flow from the outer surfaces of the membranes to the inner surfaces of the membranes.

[0009] In another embodiment of the invention, a method of cleaning hollow fiber membranes comprises (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, wherein the hollow fiber membrane has a bore, an inner surface, and an outer surface, (2) providing a first pressure to the inner surface of the hollow fiber membrane through the retentate outlet and passing the fluid along the bore of at least one hollow fiber membrane to provide a retentate and a permeate, (3) applying a second pressure to the outer surface of the hollow fiber membrane, and (4) reducing the first pressure while maintaining or increasing the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the hollow fiber membrane.

[0010] In another embodiment of the invention, a method of cleaning hollow fiber membranes comprises (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least two hollow

fiber membranes disposed in the housing, each hollow fiber membrane having a bore, an inner surface and an outer surface, (2) passing the fluid along the bores of at least two membranes to provide retentate flow along the inner surfaces of the membranes at an initial retentate flow rate and permeate flow from the inner surfaces to the outer surfaces of the membranes at an initial permeate flow rate, (3) blocking permeate flow, (4) restricting retentate flow to increase pressure on the inner surface of the membranes, (5) re-establishing retentate flow toward the initial retentate flow rate to release the increase in the pressure on the inner surfaces of the membranes and pass a portion of permeate from the outer surfaces to the inner surfaces of the membranes and pass the portion of permeate and the retentate along the inner surfaces and through the retentate outlet; and (6) repeating (4) and (5) two or more times.

[0011] In another embodiment, a method of cleaning hollow fiber membranes comprises (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, each hollow fiber membrane having a bore, an inner surface and an outer surface, (2) providing a first pressure on the inner surface of the hollow fiber membrane and passing the fluid along the bore of at least one membrane to provide retentate flow along the inner surface of the membrane and permeate flow from the inner surface to the outer surface, (3) applying a second pressure to the outer surface of the hollow fiber membrane, (4) reducing the first pressure while maintaining or increasing the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the hollow fiber membrane, and (5) repeating (3) and (4) two or more times.

[0012] In another embodiment, a hollow fiber filtration system is provided, the system comprising at least one hollow fiber filtration module including a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, the hollow fiber membrane having a bore, an inner surface, and an outer surface, wherein the system is arranged to direct feed fluid into the hollow fiber filtration module and pass the fluid into the bore of at least one hollow fiber membrane to provide a retentate flowing along the inner surface of the membrane and a permeate flowing from the inner surface of the membrane to the outer surface, a first fluid flow control device communicating with the retentate outlet, the first fluid flow control device being adapted to provide a first pressure to the inner surface of the membrane through the retentate outlet, and a second fluid flow control device communicating with the permeate outlet, the second fluid flow control device being adapted to apply a second pressure to the outer surface of the hollow fiber membrane through the permeate outlet, wherein the system is arranged to allow the first pressure and the second

pressure to be controlled such that, when desired, the first pressure is less than the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the membrane.

#### BRIEF DESCRIPTION OF THE DRAWINGS

- [0013] Figure 1 is a cross-sectional view of an illustrative hollow fiber filtration module.
- [0014] Figure 2 is a schematic of a fluid processing system according to an embodiment of the invention.
- [0015] Figure 3 is a schematic of a fluid processing system according to another embodiment of the invention.

#### DETAILED DESCRIPTION OF THE INVENTION

[0016] The present invention is suitable for use in any application involving hollow fiber membranes and tangential flow filtration wherein cleaning of the hollow fiber membranes is desirable. Applications wherein cleaning may be desirable include, for example, the electronics, pharmaceutical and/or food and beverage industries, wherein a variety of fluids are processed, e.g., to generate ultrapure water sources for the electronics and pharmaceutical industries, to separate at least one of cells, cell products, molecules and/or species of interest in the pharmaceutical industries, and to treat aqueous fluids in the food and beverage, and pulp and paper industries. It may be desirable to clean hollow fibers used to treat source water, such as municipal drinking water, water from natural sources such as lakes, rivers, reservoirs, surface water, ground water and storm water runoff, or industrial source water, or wastewater, such as industrial wastewater or municipal wastewater. Source water may also include treated wastewater which has, for example, been purified after industrial use.

[0017] In an embodiment, the invention provides a method of cleaning hollow fiber membranes comprising (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, wherein each hollow fiber membrane has a bore, an inner surface, and an outer surface, (2) providing a first pressure to the inner surface of the hollow fiber membrane through the retentate outlet and passing the fluid along the bore of at least one hollow fiber membrane to provide retentate flow along the inner surface of the membrane and permeate flow from the inner surface to the outer surface of the membrane, (3) increasing the first pressure, (4) applying a second pressure to the outer

surface of the hollow fiber membrane through the permeate outlet, and (5) releasing the increase in the first pressure while maintaining or increasing the second pressure to pass permeate from the outer surface to the inner surface of the hollow fiber membrane(s). In some embodiments, the method includes increasing the first pressure until the pressure is at least about 0.05 MPa before releasing the increase in the first pressure. Preferably, the hollow fiber filtration module comprises two or more hollow fiber membranes, and the method includes increasing the first pressure applied to the inner surface of each membrane, applying a second pressure to the outer surface of each membrane, and releasing the increase in the first pressure while maintaining or increasing the second pressure to pass permeate from the outer surface to the inner surface of each membrane. In some embodiments, the method includes increasing the first pressure and releasing the increase in the first pressure two or more times while continuously maintaining the second pressure to pass permeate from the outer surface to the inner surface of each membrane.

**[0018]** In another embodiment of the invention, a method of cleaning hollow fiber membranes comprises directing fluid into a hollow fiber filtration module including a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least two hollow fiber membranes, wherein each hollow fiber membrane has a bore, an inner surface, and an outer surface, passing fluid into the bores of at least two hollow fiber membranes to provide retentate flow along the inner surfaces of the membranes and permeate flow from the inner surfaces of the membranes to the outer surfaces, blocking the permeate flow, restricting the retentate flow, and unrestricting the retentate flow to reverse permeate flow from the outer surfaces of the membranes to the inner surfaces of the membranes. In some embodiments, fluid is passed into the bores of at least two hollow fiber membranes to provide retentate flow and permeate flow while maintaining a retentate flow rate at an average velocity of about 0.7 m/s or less. In other embodiments, the fluid is passed into the bores of at least two hollow fiber membranes to provide retentate flow and permeate flow while maintaining a retentate flow rate at an average velocity in the range of about 2 - 7 m/s or less.

**[0019]** In another embodiment of the invention, a method of cleaning hollow fiber membranes comprises (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, wherein the hollow fiber membrane has a bore, an inner surface, and an outer surface, (2) providing a first pressure to the inner surface of the hollow fiber membrane through the retentate outlet and passing the fluid along the bore of at least one hollow fiber membrane to provide retentate flow along the inner surface of the membrane and permeate flow from the inner surface to the outer surface of the membrane,

(3) applying a second pressure to the outer surface of the hollow fiber membrane, and (4) reducing the first pressure while maintaining or increasing the second pressure to pass permeate (i.e., reverse the flow of permeate) from the outer surface to the inner surface of the hollow fiber membrane. Preferably, the hollow fiber filtration module comprises two or more hollow fiber membranes, and the method includes applying the second pressure to the outer surface of each membrane, and reducing the first pressure while maintaining or increasing the second pressure to pass permeate from the outer surface to the inner surface of each membrane. In some embodiments, the method includes providing a first pressure and reducing the first pressure two or more times while continuously maintaining the second pressure to pass permeate from the outer surface to the inner surface of each membrane.

**[0020]** In another embodiment of the invention, a method of cleaning hollow fiber membranes comprises (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least two hollow fiber membranes disposed in the housing, each hollow fiber membrane having a bore, an inner surface and an outer surface, (2) passing the fluid along the bores of at least two membranes to provide retentate flow along the inner surfaces of the membranes at an initial retentate flow rate and permeate flow from the inner surfaces to the outer surfaces of the membranes at an initial permeate flow rate, (3) blocking permeate flow, (4) restricting retentate flow to increase pressure on the inner surface of the membranes, (5) re-establishing retentate flow toward the initial retentate flow rate to release the increase in the pressure on the inner surfaces of the membranes and pass a portion of permeate from the outer surfaces to the inner surfaces of the membranes and pass the portion of permeate and the retentate along the inner surfaces and through the retentate outlet; and (6) repeating (4) and (5) two or more times.

**[0021]** In another embodiment, a method of cleaning hollow fiber membranes comprises (1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, each hollow fiber membrane having a bore, an inner surface and an outer surface, (2) providing a first pressure on the inner surface of the hollow fiber membrane and passing the fluid along the bore of at least one membrane to provide retentate flow along the inner surface of the membrane and permeate flow from the inner surface to the outer surface, (3) applying a second pressure to the outer surface of the hollow fiber membrane, (4) reducing the first pressure while maintaining or increasing the

second pressure to reverse the flow of permeate from the outer surface to the inner surface of the hollow fiber membrane, and (5) repeating (3) and (4) two or more times.

**[0022]** In another embodiment, a hollow fiber filtration system is provided, the system comprising at least one hollow fiber filtration module including a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, the hollow fiber membrane having a bore, an inner surface, and an outer surface, wherein the system is arranged to direct feed fluid into the hollow fiber filtration module and pass the fluid into the bore of at least one hollow fiber membrane to provide a retentate flowing along the inner surface of the membrane and a permeate flowing from the inner surface of the membrane to the outer surface, a first fluid flow control device communicating with the retentate outlet, the first fluid flow control device being adapted to provide a first pressure to the inner surface of the membrane through the retentate outlet, and a second fluid flow control device communicating with the permeate outlet, the second fluid flow control device being adapted to apply a second pressure to the outer surface of the hollow fiber membrane through the permeate outlet, wherein the system is arranged to allow the first pressure and the second pressure to be controlled such that, when desired, the first pressure is less than the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the membrane.

**[0023]** In embodiments of the invention, a fluid is directed into a hollow fiber filtration module to be processed and the hollow fiber membranes in the module are subsequently cleaned. Typically, a feed fluid is directed into a hollow fiber filtration module to be processed, e.g., to provide a retentate and a permeate, and subsequently a cleaning fluid is directed into the hollow fiber filtration module to clean the membranes. Generally, the cleaning fluid comprises a fluid other than the feed fluid, although in some embodiments the cleaning fluid may comprise the feed fluid.

**[0024]** Using the exemplary module illustrated in Figure 1 for reference, a typical hollow fiber filtration module 10 comprises a housing 12, a feed fluid inlet 14, a retentate outlet 16, a permeate outlet 18, and a plurality of hollow fiber membranes 20 (i.e., two or more hollow fiber membranes; module with a single hollow fiber membrane not shown) disposed in the housing 12 between the inlet 14 and the outlets 16 and 18. The housing includes a first fluid flow path 22 extending between the feed inlet 14 and the retentate outlet 16 and a second fluid flow path 24 extending between the feed inlet 14 and the permeate outlet 18. The hollow fiber membranes 20 are disposed in the housing 12 tangentially to the first fluid flow path 22 and across the second fluid flow path 24. The hollow fibers 20 each include a bore 26, an inner (or upstream) surface 28, and an outer (or

downstream) surface 30. A fluid is directed into the hollow fiber filtration module 10 through the feed inlet 14 and through the bores 26 of the hollow fiber membranes 20. A first pressure is provided on the inner surface 28 of the hollow fiber membrane and retentate passes tangentially to the inner surface 28 of each membrane (i.e., along the first fluid flow path 22), and a permeate or filtrate passes from the inner surface 28 of each membrane to the outer surface 30 (i.e., along the second fluid flow path 24).

**[0025]** In some embodiments, a hollow fiber filtration module 10 may include one or more additional fluid ports. For example, in the embodiment illustrated in Figure 1, the hollow fiber filtration module 10 includes one additional fluid port 32 communicating with the outer surface 30 of the hollow fiber membranes 20. The additional fluid port 32 may be used to introduce fluid to the hollow fiber filtration module. For example, in conventional hollow fiber module cleaning methods, a backwash fluid can be introduced through the additional fluid port 32.

**[0026]** In accordance with the invention, one or more hollow fiber filtration modules are utilized in a hollow fiber filtration system, and, in a typical embodiment, a first pressure is applied to the inner surface 28 of the hollow fiber membranes through the retentate outlet 16, and a second pressure is applied to the outer surface 30 of the hollow fiber membranes through the permeate outlet 18. As will be described in more detail below, the first and second pressures can be adjusted so that, when desired, the second pressure is greater than the first pressure, and permeate is passed from the outer surface 30 to the inner surface 28, i.e., the flow of permeate is reversed through the surfaces of the membrane. Subsequently, permeate passing from the outer surface to the inner surface is passed along the inner surface and through the retentate outlet 16.

**[0027]** For example, in some embodiments, the first pressure (e.g., at an initial value) can be increased to a higher pressure, and the increase in the first pressure is released or substantially released (e.g., toward the initial value) while maintaining or increasing the second pressure to pass permeate from the outer surface 30 to the inner surface 28 of the hollow fiber membranes. The second pressure is subsequently released. In some embodiments, the second pressure is not released until the first pressure is increased and released or substantially released, two or more times, to further enhance the cleaning of the membranes 20.

**[0028]** In other embodiments, the first pressure is decreased from the initial value, and the second pressure is applied to the outer surface 30 of the hollow fiber membranes, and the second pressure is maintained or increased to pass permeate from the outer surface 30 to the inner surface 28 of the hollow fiber membranes. Subsequently, the second pressure is

released and the first pressure is increased, e.g., returned, or substantially returned, to the initial value.

**[0029]** Without being bound to any particular theory, it is believed that, for those embodiments wherein the first pressure is increased while applying a second pressure, this results in an expansion of the housing (and/or the inner diameter of the membranes) and an increase in the module volume. Releasing or reducing the increase in the first pressure then produces a contraction of the housing (and/or the interior diameter of the membranes), providing a force for flowing the permeate from the outer surface to the inner surface of the membranes.

**[0030]** Figure 2 shows an illustrative hollow fiber filtration system 100 according to an embodiment of the invention including one hollow fiber filtration module 10 as described above. The hollow fiber filtration system 100 includes a plurality of conduits providing fluid communication between the components of the system, and preferably includes a retentate flow control device 112 disposed at or downstream of the retentate outlet 16 for providing a first pressure on the inner surface 28 of the hollow fiber membrane, and a permeate flow control device 110 disposed at or downstream of the permeate outlet 18 for applying a second pressure to the outer surface 30 of the hollow fiber membranes. Preferably, the permeate flow control device 110 and the retentate flow control device 112 comprise adjustable valves.

**[0031]** The illustrated hollow fiber filtration system 100 also includes an optional cleaning fluid inlet flow control device 116 for introducing a cleaning fluid into the system 100 and a cleaning fluid outlet flow control device 118 for removing cleaning fluid from the system (or for returning it to the container including the cleaning fluid). In some of these embodiments, the system 100 also includes a cleaning fluid container or reservoir 120a communicating with the system 100 via the cleaning fluid inlet flow control device 116 and cleaning fluid outlet flow control device 118 and the hollow fiber filtration module 10 via the feed inlet 14 and the retentate outlet 16. However, as will be explained in more detail below, embodiments of the invention do not require the use of cleaning fluid containers and/or cleaning fluid inlet and/or outlet flow control devices. Alternatively, for example, one or more of the permeate and retentate flow control devices can comprise multi-way valves (e.g., 3-way valves) to allow cleaning fluid flow and permeate and/or retentate flow, when desired.

**[0032]** The illustrated hollow fiber filtration system 100 also includes a feed container or reservoir 120 disposed upstream of the feed fluid inlet 14 (and the cleaning fluid inlet flow control device 116), and a permeate container or reservoir 122, wherein the hollow

fiber filtration module 10 fluidly communicates with the feed container 120 via a feed fluid conduit 121 and the feed inlet 14 and the permeate container 122 via a permeate conduit 123 and the permeate outlet 18. In the illustrated system, the retentate outlet 16 also fluidly communicates, via a retentate conduit 125, with the feed container 120, such that retentate may be recirculated to the hollow fiber filtration module 10 as feed fluid. Alternatively, the system may include a separate retentate container or reservoir (not shown) that may communicate with the hollow fiber filtration module 10 via the retentate outlet 16.

[0033] In some embodiments, the feed container 120 may be suitable for containing the cleaning fluid, or the feed container 120 can be replaced with a cleaning fluid container 120a (shown in dotted lines) when cleaning is carried out. Alternatively, for example, both feed container 120 and cleaning fluid container 120a can be used, e.g., cleaning fluid container 120a can be placed in fluid communication with the system 100, for example, via a cleaning fluid inlet conduit 126 and cleaning fluid inlet control device 116 and a cleaning fluid return conduit 128 and cleaning fluid outlet control device 118.

[0034] Since, in some embodiments, a portion of cleaning fluid permeate can be produced that is not subsequently passed in a reverse direction through the membrane surfaces, the permeate container 122 may also be suitable for containing permeate produced from the cleaning fluid, or the permeate container can be replaced with a cleaning fluid permeate container 122a (shown in dotted lines). Alternatively, both permeate container 122 and cleaning fluid permeate container 122a can be used, e.g., connected to the system via a suitable fluid flow control device. Typically, in those embodiments wherein the feed container 120 and the permeate container 122 are replaced by a cleaning fluid container 120a and a cleaning fluid permeate container 122a (in contrast with, for example, leaving containers 120 and 122 attached to the system during cleaning), the system does not include or does not require the use of the cleaning fluid inlet flow control device 116 and/or the cleaning fluid outlet flow control device 118.

[0035] Additionally, the system may include additional components, and in the embodiment illustrated in Figure 2, the system includes a feed flow control device 114 disposed at or upstream of the feed inlet 14, a pump 124 preferably disposed between the feed container 120 and the feed flow control device 114. In some embodiments, the fluid processing system 100 may also include a pressure storage reservoir such as an accumulator (not shown), e.g., disposed downstream of the permeate flow control device 110 for ease in providing the second pressure to the hollow fiber membranes 20.

[0036] In operation of the system, the cleaning fluid is directed into the hollow fiber filtration module 10 and passed into and along the bores 26 of the hollow fiber membranes

to provide a retentate and a permeate. The cleaning fluid can be provided to the module 10 from the feed container 120 or the cleaning fluid container 120a, and the cleaning fluid retentate can be returned to the feed container or the cleaning fluid container. In some embodiments, the cleaning fluid is passed from cleaning fluid container 120a via cleaning fluid inlet conduit 126 to cleaning fluid inlet flow control device 116 and to the module 10, and the cleaning fluid retentate is passed from the retentate outlet 16 via retentate conduit 125 to cleaning fluid outlet flow control device 118 and to the cleaning fluid container 120a via cleaning fluid return conduit 128. As described above, a first pressure is provided to the inner surface 28 of the hollow fiber membranes, preferably through the retentate outlet 16 and a second pressure is applied to the outer surface 30 of the hollow fiber membranes, preferably through the permeate outlet 18, and the first and second pressures are adjusted as described above so that a portion of the permeate passing from the inner surface 28 to the outer surface 30 is passed in the reverse direction, i.e., from the outer surface 30 to the inner surface 28. Preferably, the portion of reversed flow permeate, i.e., the permeate passing from the outer surface 30 to the inner surface 28 of the hollow fibers, and the retentate, are passed along the inner surface 28 and through the retentate outlet 16.

[0037] Typically, the first pressure is increased from an initial value and the second pressure is applied by at least partially closing the retentate flow control device 112, and closing the permeate flow control device, 110, respectively. The increase in the first pressure is preferably released while maintaining or increasing the second pressure to reverse the flow of permeate through the hollow fiber membranes, i.e., to pass permeate from the outer surface 30 to the inner surface 28 of the hollow fiber membranes. The first pressure may be released at any suitable time and/or pressure, for example, in some embodiments, the first pressure is released when the pressure (from an initial value of, for example, about .01 MPa) reaches from about 0.05 to about 0.6 MPa. The pressure level at which the first pressure is released is preferably chosen in accordance with the particular module being cleaned, e.g., the first pressure is released before exceeding a threshold pressure for the particular module. For example, in some embodiments, the first pressure is released when the pressure reaches about 0.2 MPa or about 0.3 MPa.

[0038] In another embodiment, the first pressure is decreased from an initial value and the second pressure is applied by further opening the retentate flow control device 112, and closing the permeate flow control device, 110, respectively. The first pressure is preferably decreased while maintaining or increasing the second pressure to reverse the flow of permeate through the hollow fiber membranes, i.e., to pass permeate from the outer surface 30 to the inner surface 28 of the hollow fiber membranes. The initial value for the first

pressure can be any suitable pressure as is known in the art, for example, in some embodiments the initial value can be a pressure of about 0.2 MPa or more, e.g., from about 0.3 MPa to about 0.5 MPa. The first pressure can be decreased to any suitable value, e.g., about 0.01 MPa to about 0.15 MPa.

[0039] In accordance with these embodiments, the portion of permeate passing from the outer surface 30 to the inner surface 28 of the hollow fibers and the retentate is preferably passed along the inner surface 28 and through the retentate outlet 16.

[0040] In some embodiments, the fluid is passed into the bores 26 of the hollow fiber membrane while maintaining the retentate flow at a desired average velocity. For example, the fluid may be passed into the bores 26 while maintaining the retentate flow rate at an average velocity of about 0.7 m/s or less. In other embodiments, the retentate flow rate is much higher, for example, the retentate flow rate may be maintained at an average velocity of about 2 m/s to about 7 m/s. The flow rate of retentate may be maintained at the desired average velocity by a variety of means. For example, in some embodiments, the flow rate of retentate may be controlled using a flow control device 112, such as a valve (preferably a variable flow valve), disposed at or near the retentate outlet 16 or by using pump 124 to control the feed fluid flow rate into the feed inlet 14 and the membrane bore(s) 26.

[0041] In some embodiments, to more effectively clean the hollow fiber membranes, the operations described above may be repeated one or more times. For example, the first pressure may be increased from an initial value and the first pressure can be subsequently reduced to the initial value or substantially the initial value, two, three, four, five, or even more, times, preferably while continuously maintaining the second pressure. Alternatively, the first pressure can be decreased from an initial value and the first pressure can be subsequently increased to the initial value or substantially the initial value, two, three, four, five, or even more, times, preferably while continuously maintaining the second pressure.

[0042] In other embodiments, the second pressure is not continuously maintained, e.g., it is repeatedly released, increased, and released, until cleaning is completed.

[0043] A variety of factors may affect the number of repetitions utilized, if any, for example, the degree of fouling, the nature of the feed fluid, and/or the characteristics of the membranes.

[0044] Some preferred embodiments of systems according to the invention include a plurality of hollow fiber filtration modules, and any number of modules can be utilized in accordance with the invention, for example, 2, 3, 4 or 5 modules, or more. Figure 3 illustrates an embodiment of a fluid processing system 200 including four hollow fiber modules 10a-10d. The embodiment of the fluid processing system 200 illustrated in Figure

3 includes many of the components shown in the embodiment of the system 100 shown in Figure 2. For example, Figure 3 includes a feed container or reservoir 120 and an alternate cleaning fluid container 120a, and a permeate container or reservoir 122 and an alternate cleaning fluid permeate container 122a, wherein each of the hollow fiber filtration modules 10a-10d includes a feed inlet (14a-14d, respectively), retentate outlet (16a-16d, respectively), and a permeate outlet (18a-18d, respectively). The system 200 also includes feed fluid conduit 121 and retentate conduit 125 communicating with the feed container 120 (or cleaning fluid container 120a) and the permeate conduit 123 communicating with the permeate container 122 (or cleaning fluid permeate container 122a). Each of the hollow fiber filtration modules 10a-10d communicates with the feed fluid conduit 121 via the feed inlet (14a-14d, respectively) and a module feed fluid conduit (121a-121d, respectively), and the permeate conduit 123 via the permeate outlet (18a-18d, respectively) and a module permeate conduit (123a-123d, respectively). Each of the hollow fiber filtration modules 10a-10d communicates with the retentate conduit 125 via the retentate outlet (16a-16d, respectively) and a module retentate conduit (125a-125d, respectively). The feed fluid conduit 121 and module feed fluid conduits 121a-121d, the permeate conduit 123 and module permeate conduits 123a-123d, and the retentate conduit 125 and module retentate conduits 125a-125d may be connected using standard connectors and/or connections known in the art, such as T-joints, welds, and the like. The system 200 also includes a cleaning fluid inlet conduit 126 and a cleaning fluid inlet flow control device 116 and a cleaning fluid return conduit 128 and a cleaning fluid outlet flow control device 118 for optionally introducing cleaning fluid into and removing cleaning fluid from the system 200, as well as a pump 124 preferably disposed between the feed container 120 and the feed flow control device 114 for providing a flow of fluid to the modules 10a-10d.

[0045] In the illustrated embodiment, the multi-module system 200 also includes at least one permeate flow control device for applying a second pressure to the outer surface 30 of the hollow fiber membranes and at least one retentate flow control device for providing a first pressure to the inner surface of the hollow fiber membranes. In this illustrated embodiment, the permeate flow control device 110 comprising a permeate valve disposed downstream of all of the permeate outlets 18a-18d applies the pressure to the outer surface of the membranes 30 for all of the modules 10a-10d. Similarly, the retentate flow control device comprising a retentate flow control device 112 disposed downstream of all of the retentate outlets 16a-16d applies the pressure to the inner surface 28 of the membranes for all of the modules 10a-10d. However, if desired, the system 200 can include a plurality of permeate flow control devices for applying the pressure to the outer surfaces 30 of the

membranes and a plurality of retentate flow control devices for applying the pressure to the inner surface 28 of the membranes. Illustratively, a separate permeate flow control device and/or separate retentate flow control device can be utilized with each module, or, for example, a plurality of permeate and/or retentate flow control devices, wherein each flow control device applies pressure to two or more modules, can be utilized.

[0046] Embodiments of the system can include a variety of configurations.

Additionally, a variety of fluid processing system components, e.g., one or more hollow fiber filtration modules, hollow fiber membranes (such as polymer membranes, skinned (including single-skinned and double-skinned) and non-skinned membranes), tubing, flow control devices (such as valves and pumps), feed and permeate containers, and pressure storage reservoirs, are suitable for use in accordance with the invention and are commercially available. Illustratively, the particular module(s), membrane(s), number of membranes, pore size(s) or molecular weight cut off(s) utilized will depend on, for example, the feed fluid being processed and/or the desired product, as is known in the art.

[0047] A variety of flow control devices are suitable for use in the invention, including clamps, ball valves, diaphragm valves, needle valves, and pumps. Flow control devices can be variable flow, i.e., capable of providing varying flow rates, and binary on/off that open to permit flow and close to block flow. Suitable pumps include, but are not limited to, vane pumps, tubing pumps, rotary pumps, and diaphragm pumps, and may be chosen based upon, for example, the desired flow rate.

[0048] The system can include a plurality of types of valves, for example a first variable flow valve (e.g., valve 112) arranged to provide, increase and/or reduce the first pressure to the inner surface of the membranes through the retentate outlet, a second variable flow valve (e.g., valve 110) arranged to apply and release the second pressure to the outer surfaces of the membranes through the permeate outlet, and a binary on/off valve (e.g., valve 114) arranged to permit feed flow into the module when open and to block flow when closed.

[0049] A variety of fluids may be suitable for cleaning the hollow fiber membranes in accordance with the present invention. For example, a non-chemical cleaning agent (e.g., water) and/or a chemical cleaning agent (e.g., a caustic cleaning agent) may be suitable. In some applications, the membranes are cleaned a number of times using a non-chemical treatment before a chemical treatment is utilized. For example, in one embodiment, over a 24 hour period, the membranes may be cleaned non-chemically several times, and cleaned chemically once. In another protocol, the membranes may be cleaned non-chemically at least once a day, and cleaned chemically once a week, or once every 30 days, for example.

[0050] In other embodiments, the cleaning fluid may comprise feed fluid. Utilizing feed fluid as the cleaning fluid offers advantages in some applications, such as minimizing cleaning time and preventing the introduction of foreign fluids into the system.

[0051] After cleaning, filtration is continued for a desired period of time, or, for example, until the flux decreases to a predetermined value or range or the differential pressure increases to a predetermined value or range. Filtration is then stopped, and the membranes are cleaned. After cleaning, filtration is resumed, and additional fluid to be treated is passed through the membranes until cleaning is again appropriate.

[0052] The following examples further illustrate the invention but, of course, should not be construed as in any way limiting its scope.

[0053] **EXAMPLE**

[0054] This example demonstrates the regeneration of hollow fiber membranes in accordance with an embodiment of the invention.

[0055] A fluid processing system is arranged as generally shown in Figure 2. The system includes a MICROZA® Lab Module (Asahi Chemical Industry Co., Ltd., Japan; Model XULP-143) with hollow fiber membranes having a pore size of 0.45  $\mu\text{m}$  and a total membrane area of 0.2  $\text{m}^2$ .

[0056] After twice concentrating a 5L solution containing 10% bakery yeast cells (operated at a flow rate of 3L/min), the system is cleaned. The permeate valve downstream of the permeate outlet is closed, and the cleaning fluid, water, is flowed through the module to provide a retentate flow rate of about 2 L/min and a retentate pressure of about 0.01 MPa. The retentate valve downstream of the retentate outlet is then partially closed until the retentate pressure rises to about 0.15 MPa, at which time the retentate valve is quickly opened, producing a backflow of permeate from outer surface of the membranes to the inner surface of the membranes. The retentate valve is again partially closed until the retentate pressure rises to about 0.15 MPa at which time the retentate valve is opened producing the backflow of permeate. After the retentate valve has been partially closed and quickly opened 5 times, the permeate flux is then measured, and determined to be 26 liters/ $\text{m}^2/\text{hr}$  (LMH).

[0057] Another fluid processing system is arranged including a MICROZA® Lab Module, and a solution of bakery yeast cells is concentrated as described above. This system is cleaned by closing the permeate valve and using a reverse cleaning line and an additional pump to introduce the cleaning fluid through an additional port fluidly communicating with the outer surfaces of the membranes at a flow rate of about 4L/min for

approximately 3 seconds. The retentate valve remains open during the cleaning procedure. The permeate flux is then measured and determined to be 23 LMH.

[0058] This example shows membranes can be regenerated (cleaned) in accordance with embodiments of the invention without requiring a separate cleaning line.

[0059] All references, including publications, patent applications, and patents, cited herein are hereby incorporated by reference to the same extent as if each reference were individually and specifically indicated to be incorporated by reference and were set forth in its entirety herein.

[0060] The use of the terms “a” and “an” and “the” and similar referents in the context of describing the invention (especially in the context of the following claims) are to be construed to cover both the singular and the plural, unless otherwise indicated herein or clearly contradicted by context. Recitation of ranges of values herein are merely intended to serve as a shorthand method of referring individually to each separate value falling within the range, unless otherwise indicated herein, and each separate value is incorporated into the specification as if it were individually recited herein. All methods described herein can be performed in any suitable order unless otherwise indicated herein or otherwise clearly contradicted by context. The use of any and all examples, or exemplary language (e.g., “such as”) provided herein, is intended merely to better illuminate the invention and does not pose a limitation on the scope of the invention unless otherwise claimed. No language in the specification should be construed as indicating any non-claimed element as essential to the practice of the invention.

[0061] Preferred embodiments of this invention are described herein, including the best mode known to the inventors for carrying out the invention. Of course, variations of those preferred embodiments will become apparent to those of ordinary skill in the art upon reading the foregoing description. The inventors expect skilled artisans to employ such variations as appropriate, and the inventors intend for the invention to be practiced otherwise than as specifically described herein. Accordingly, this invention includes all modifications and equivalents of the subject matter recited in the claims appended hereto as permitted by applicable law. Moreover, any combination of the above-described elements in all possible variations thereof is encompassed by the invention unless otherwise indicated herein or otherwise clearly contradicted by context.

## WHAT IS CLAIMED IS:

1. A method of cleaning hollow fiber membranes comprising:

directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, wherein the hollow fiber membrane has a bore, an inner surface, and an outer surface;

providing a first pressure to the inner surface of the hollow fiber membrane through the retentate outlet and passing the fluid along the bore of at least one hollow fiber membrane to provide a retentate and a permeate;

increasing the first pressure;

applying a second pressure to the outer surface of the hollow fiber membrane through the permeate outlet; and

releasing the increased first pressure while maintaining or increasing the second pressure to pass permeate from the outer surface to the inner surface of the hollow fiber membrane.

2. The method of claim 1, comprising releasing the increased first pressure while maintaining the second pressure to pass permeate from the outer surface to the inner surface of the hollow fiber membrane.

3. The method of claim 1 or 2, including increasing the first pressure until the pressure is about 0.05 MPa before releasing the increased first pressure.

4. A method of cleaning hollow fiber membranes comprising:

(1) directing fluid into a hollow fiber filtration module including a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least two hollow fiber membranes, wherein each hollow fiber membrane has a bore, an inner surface, and an outer surface;

(2) passing fluid into the bores of at least two hollow fiber membranes to provide retentate flow along the inner surfaces of the membranes and permeate flow from the inner surfaces of the membranes to the outer surfaces;

(3) blocking the permeate flow;

(4) restricting the retentate flow; and

(5) unrestricting the retentate flow to reverse permeate flow from the outer surfaces of the membranes to the inner surfaces of the membranes.

5. The method of claim 4 wherein fluid is passed into the bores of at least two hollow fiber membranes to provide retentate flow and permeate flow while maintaining a retentate flow rate at an average velocity of about 0.7 m/s or less.

6. The method of claim 4 wherein fluid is passed into the bores of at least two hollow fiber membranes to provide retentate flow and permeate flow while maintaining a retentate flow rate at an average velocity of about 7 m/s or less.

7. The method of any one of claims 4-6, wherein restricting and unrestricting retentate flow comprises operating a valve downstream of the retentate outlet.

8. The method of any one of claims 4-7, wherein blocking permeate flow comprises closing a valve downstream of the permeate outlet:

9. The method of any one of claims 4-6, wherein restricting and unrestricting retentate flow comprises at least partially closing and opening the retentate outlet.

10. The method of any one of claims 4-7, wherein blocking permeate flow comprises closing the permeate outlet.

11. The method of any one of claims 4-10, wherein restricting the retentate flow increases pressure on the inner surface of the membranes and unrestricting the retentate flow comprises reestablishing retentate flow toward an initial retentate flow rate to release the increase in the pressure on the inner surfaces of the membranes and pass a portion of permeate and the retentate along the inner surfaces and through the retentate outlet, the method further comprising repeating (4) and (5) two or more times.

12. A method of cleaning hollow fiber membranes comprising:

(1) directing a fluid into a hollow fiber filtration module comprising a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing, wherein the hollow fiber membrane has a bore, an inner surface, and an outer surface;

(2) providing a first pressure to the inner surface of the hollow fiber membrane through the retentate outlet and passing the fluid along the bore of at least one hollow fiber membrane to provide a retentate and a permeate;

(3) applying a second pressure to the outer surface of the hollow fiber membrane; and

(4) reducing the first pressure while maintaining or increasing the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the hollow fiber membrane.

13. The method of claim 12, comprising reducing the first pressure while maintaining the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the hollow fiber membrane.

14. The method of claim 12 or 13 wherein providing the first pressure comprises providing a pressure of about 0.2 MPa or more.

15. The method of any one of claims 12-14 wherein applying the second pressure to the outer surface of the hollow fiber membrane comprises closing a valve downstream of the permeate outlet.

16. The method of any one of claims 12-15 further comprising repeating (3) and (4) two or more times.

17. A hollow fiber filtration system comprising:  
at least one hollow fiber filtration module including a housing, a feed fluid inlet, a retentate outlet, a permeate outlet, and at least one hollow fiber membrane disposed in the housing;  
the hollow fiber membrane having a bore, an inner surface, and an outer surface;  
the system being arranged to direct feed fluid into the hollow fiber filtration module and pass the fluid into the bore of at least one hollow fiber membrane to provide a retentate flowing along the inner surface of the membrane and a permeate flowing from the inner surface of the membrane to the outer surface;  
a first fluid flow control device communicating with the retentate outlet, the first fluid flow control device being adapted to provide a first pressure to the inner surface of the membrane through the retentate outlet; and

a second fluid flow control device communicating with the permeate outlet, the second fluid flow control device being adapted to apply a second pressure to the outer surface of the hollow fiber membrane through the permeate outlet;

wherein the system is arranged to allow the first pressure and the second pressure to be controlled such that the first pressure is less than the second pressure to reverse the flow of permeate from the outer surface to the inner surface of the membrane.

18. The system of claim 17, wherein the first fluid flow control device and the second fluid flow control device each comprise an adjustable valve.

19. The system of claim 18, wherein the first fluid flow control device is arranged to allow or prevent fluid flow through the retentate outlet.

20. The system of any one of claims 17-19, wherein the second fluid flow control device is arranged to allow or prevent fluid flow through the permeate outlet.

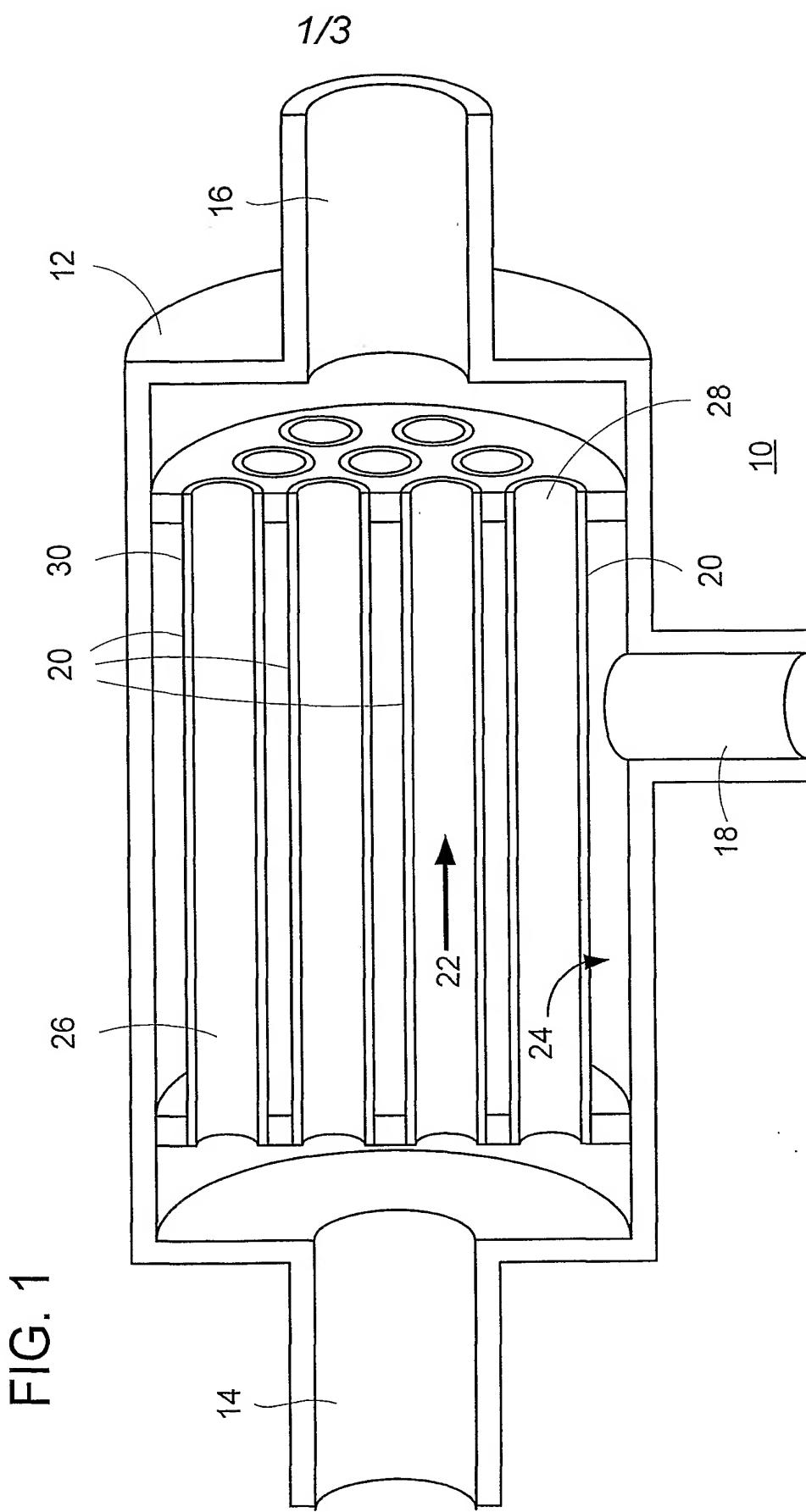


FIG. 1

2/3

FIG. 2

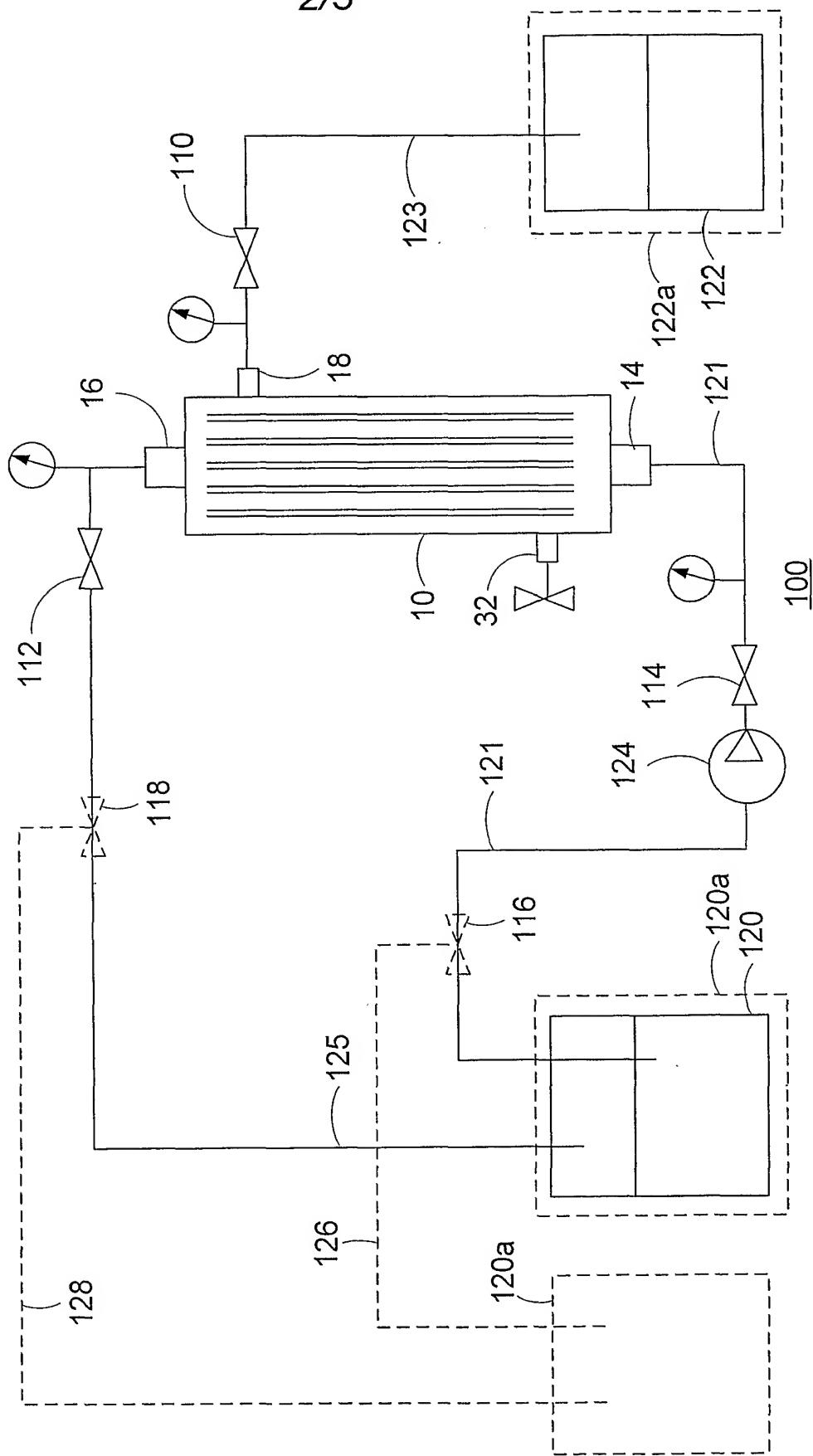


FIG. 3

